

\* NOTICES \*

**BEST AVAILABLE COPY**

JPO and NCIP are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

**CLAIMS**

---

**[Claim(s)]**

[Claim 1] The high activity calcium-oxide porous body which consists of the granulated body baking object of a calcium hydroxide with the specific surface area of at least 5m<sup>2</sup>/g, and the particle size of at least 1mm, or a calcium carbonate.

[Claim 2] calcium-hydroxide powder with a particle size of 300 micrometers or less -- particle size -- the manufacture approach of the high activity calcium-oxide porous body according to claim 1 characterized by corning into 1mm granulation even if few, heating this granulated body under ordinary pressure, carrying out a temperature up, applying [ degrees C / 390-480 ] it for at least 5 minutes, and calcinating.

[Claim 3] It corns into 1mm granulation. calcium-hydroxide powder with a particle size of 300 micrometers or less -- particle size, even if few In the temperature of the arbitration within the limits of further 480-950 degrees C after heating this granulated body under ordinary pressure, and carrying out a temperature up, having applied [ degrees C / 390-480 ] it for at least 5 minutes The manufacture approach of a high activity calcium-oxide porous body according to claim 1 that CO<sub>2</sub> conversion is characterized by calcinating by the time amount within the limits which do not fall to 40% or less.

[Claim 4] calcium-carbonate powder with a particle size of 300 micrometers or less -- particle size -- the manufacture approach of the high activity calcium-oxide porous body according to claim 1 characterized by corning into 1mm granulation even if few, heating this granulated body under ordinary pressure, carrying out a temperature up, applying [ degrees C / 700-780 ] it for at least 5 minutes, and calcinating.

[Claim 5] It corns into 1mm granulation. calcium-carbonate powder with a particle size of 300 micrometers or less -- particle size, even if few In the temperature of the arbitration within the limits of further 780-950 degrees C after heating this granulated body under ordinary pressure, and carrying out a temperature up, having applied [ degrees C / 700-780 ] it for at least 5 minutes The manufacture approach of a high activity calcium-oxide porous body according to claim 1 that CO<sub>2</sub> conversion is characterized by calcinating by the time amount within the limits which do not fall to 40% or less.

---

[Translation done.]

\* NOTICES \*

**BEST AVAILABLE COPY**

JPO and NCIP are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

**DETAILED DESCRIPTION**

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the high activity calcium-oxide porous body which specific surface area is large, and grain size is moreover large, and is easy to deal with it, and its manufacture approach.

[0002]

[Description of the Prior Art] Although the calcium oxide is manufactured by usually calcinating a calcium carbonate, as for this thing, it is known that activity changes with burning temperature. That is, although what is obtained with the burning temperature near the decarboxylation temperature of a calcium carbonate is called soft-burned lime and it is rich in activity, what is further calcinated at an elevated temperature is called hard-burned lime, and its activity is low. By the crystal growth of a calcium oxide, and glow tightness, this is [ decline in opening voidage, and ] for specific surface area to fall, when it puts in another way. For example, speaking of the pure article, the opening voidage of hard-burned lime is very as small [ specific surface area ] as about 0.04m<sup>2</sup>/g about 10% to the opening voidage of soft-burned lime being about 50%, and specific surface area being about 2m<sup>2</sup>/g.

[0003] In order to obtain the calcium oxide of high activity, the attempt which makes fineness small and enlarges specific surface area has so far been made, but since there is a limit in making the powder of a calcium carbonate small, as long as a calcium carbonate is used as a raw material, specific surface area can seldom be enlarged but the largest specific surface area of the calcium-oxide powder by baking by ordinary pressure is about [ 3m ] 2/g. moreover, ["GIPUSAMU- and - lime (Gypsum & Lime)" which there are many problems on a facility and is not put in practical use although raising activity by baking under a vacuum is also proposed, No. 178, and the 31-40th page ---] .

[0004] On the other hand, the detailed powder of a calcium hydroxide is used as a raw material. Although the example which calcinated this at the temperature of 300-390 degrees C under the vacuum, and manufactured the calcium oxide powder of the large specific surface area 110-133m<sup>2</sup>/g is also known, ["a journal OBU JI American ceramic society (J. Am.Ceramic Soc.)". Though it is high activity, the particle size of 1-10 micrometers, and since the powder obtained by the 64th volume, No. 2, the 74-80page], and this approach is detailed, it is hard to deal with it and it does not escape that that field of the invention is restricted.

[0005]

[Problem(s) to be Solved by the Invention] Specific surface area is large, therefore this invention is high activity, moreover its grain size is large, and it is easy to deal with it, and it is made for the purpose of offering an available high activity calcium-oxide porous body over large range, such as a catalyst, an adsorbent for exhaust gas, heat insulating material for steel manufacture, and flux for steel manufacture.

[0006]

[Means for Solving the Problem] this invention persons came to make this invention for high activity being shown based on a header and this knowledge, although the calcium-oxide porous body calcinated and obtained at comparatively low temperature under the predetermined heating condition was a large grain size after corning the impalpable powder of a calcium hydroxide or a calcium carbonate, as a result of repeating research wholeheartedly, in order to obtain a high activity calcium oxide.

[0007] That is, this invention offers the high activity calcium-oxide porous body which consists of the granulated body baking object of a calcium hydroxide with the specific surface area of at least 5m<sup>2</sup>/g, and the particle size of at least 1mm, or a calcium carbonate.

[0008] According to this invention, such a high activity calcium-oxide porous body It corns into 1mm granulation. calcium-hydroxide powder with a particle size of 300 micrometers or less -- particle size, even if few [ whether a temperature up is carried out by heating this granulated body under ordinary pressure, applying / degrees C / 390-480 / it for at least 5 minutes, and ] It corns into 1mm granulation. or calcium-carbonate powder with a particle size of 300 micrometers or less -- particle size, even if few This granulated body can be heated under ordinary pressure, a temperature up can be carried out, applying [ degrees C / 700-780 ] it for at least 5 minutes, and it can manufacture by calcinating by the time amount within the limits to which CO<sub>2</sub> conversion does not fall to 40% or less at the temperature to 950 more degrees C if needed.

[0009] under the present circumstances, although a commercial calcium hydroxide (slaked lime) may be used for the calcium hydroxide which is boiled and is used as a raw material as it is and the hydration product of a commercial calcium oxide (calcined lime) can also be used for it, it is impure -- a part -- since the activity of the calcium oxide obtained will fall if it mixes, it is desirable to use what has purity high as much as possible. This calcium hydroxide is used as powder with a mean particle diameter of 10-300 micrometers.

[0010] Moreover, light \*\* heavy any are sufficient as a calcium carbonate, a commercial item may be used for it as it is, and what was manufactured through the carbon dioxide in the calcium-hydroxide water solution may be used for it.

performed by carrying out extrusion molding to cylindrical granulation with the minimum diameter, at least 1mm [ of 3-6mm ], for example, a diameter, and a die length of about 3-6mm using the granulating machine of common use. Under the present circumstances, as an amount of the moisture to add, 5 - 25% of the weight of the range is suitable based on the weight of a calcium hydroxide. In order to raise firmness on the occasion of this granulation, an organic binder can also be added according to a request. As this organic binder, water soluble polymer matter, such as a carboxymethyl cellulose (CMC) and polyvinyl alcohol, is used. 0.5 - 5% per weight of a calcium hydroxide of range is suitable for the addition of this organic binder.

[0012] Thus, although the obtained granulated body is subsequently calcinated, for example using an electric furnace, in the case of a calcium hydroxide, it needs to carry out the temperature up of between the range of 390-480 degrees C for at least 5 minutes in the case of a calcium carbonate, applying between the range of 700-780 degrees C for at least 5 minutes as this baking condition. When it calcinates conditions other than this, the thing of high activity more than specific-surface-area of 5m<sup>2</sup>/g cannot be obtained.

[0013] If the upper limit which is 780 degrees C is reached when 1-10-degree-C range for /is desirable, and it is a calcium hydroxide as a programming rate in this case and it is 480 degrees C and a calcium carbonate, it will be desirable to suspend heating as early as possible. Measurement of a pore distribution spectrum accepts a peak near 0.02-0.2 micrometer at this time.

[0014] Although it is necessary to continue a temperature up further in order to aim at perfect baking of a brewing raw material, it is the temperature which does not exceed 950 degrees C in this case, and it must take care that CO<sub>2</sub> conversion moreover does not become less than 40% in the case of extensive processing, and it must be performed. This extracts a sample for every fixed time amount progress, and is performed by measuring CO<sub>2</sub> conversion about the sample.

[0015] This inclination becomes remarkable, while the coagulation of the once obtained high activity calcium oxide will take place. activity will begin to fall and time amount will increase, if this heating time is too long and CO<sub>2</sub> conversion will be fallen to 40% or less.

[0016] CO<sub>2</sub> conversion in this invention contacts the mixed gas of CO<sub>2</sub> and N<sub>2</sub> by which CO<sub>2</sub> concentration is beforehand known by the sample for 1 minute in 20 degrees C, \*\* the difference of CO<sub>2</sub> concentration before and behind contact by CO<sub>2</sub> concentration before contacting, and is defined as what multiplied by 100.

[0017] Firing time can be shortened by adding the reducing agent disassembled at low temperature like a calcium fluoride on the occasion of the above-mentioned baking. Moreover, according to this invention approach, the calcium oxide adjusted to extent of a request of activity can be obtained by choosing the above-mentioned manufacture conditions.

[0018] Thus, the calcium-oxide porous body in which specific surface area usually has the high activity of 10-60m<sup>2</sup>/g more than 5m<sup>2</sup>/g is obtained as a granule of 1-6mm of diameters. If needed, this porous body is ground still more finely and various applications are presented with it.

[0019]

[Effect of the Invention] Since the calcium oxide of this invention has a large specific surface area of at least 5m<sup>2</sup>/g, shows very high activity above and is a porous body, since it can use for a catalyst, an exhaust gas adsorbent, the lagging material for steel manufacture, the flux for steel manufacture, etc., it has the advantage of being very easy to deal with it, as a product of a larger grain size than the conventional calcium oxide. Moreover, according to this invention approach, there is also an advantage that a calcium oxide with the activity according to the purpose of use can be obtained by selection of manufacture conditions.

[0020]

[Example] Next, an example explains this invention to a detail further. In addition, it asks for CO<sub>2</sub> conversion and specific surface area in each example as follows.

(1) CO<sub>2</sub> conversion; it is JIS about CO<sub>2</sub> concentration in mixture before and after inserting 5g of samples in a cylindrical reactor with a bore [ of 20mm ], and a die length of 600mm, passing the mixture of CO<sub>2</sub> gas and N<sub>2</sub> gas at 100ml a rate for /in the temperature of 20 degrees C and contacting a sample. It measured with R6124 high temperature combustion method, and calculated according to the degree type from the obtained result.

[0021]

[Equation 1]

[0022] (2) Specific surface area; after measuring 0.5g of each sample by the BET \*\*\*\* method using a mono-soap specific-surface-area measuring device (Yuasa Ionics make), the obtained measured value was doubled two and it considered as the surface area per g.

[0023] 25 % of the weight of water was added to example 1 calcium-hydroxide powder [the product made from Bell Woodwork Business, industrial use slaked lime, the purity of 95.9%, and the grain size of 300 micrometers or less], and it corned into granulation with a diameter [ of 3mm ], and a die length of 3mm using disk PERETA [Fuji Paudal Make].

[0024] When this granulated body was put into an electric furnace, it heated to 470 degrees C by part for programming-rate/of 1 degree C and it amounted to 470 degrees C, it took out from the electric furnace immediately and cooled radiationally. The heating up time from 390 degrees C to 480 degrees C in this case was 90 minutes.

[0025] When pore distribution of this thing was measured using the pore distribution meter (PORESIZER9310 by micro Medex, Inc), the highest peak was accepted in the location of 0.06 micrometers. Moreover, CO<sub>2</sub> conversion of this thing was 97%, and specific surface area was 57.67m<sup>2</sup>/g.

[0026] Example 2 calcium-carbonate powder [the BIHOKU FUNKA make, the purity of 98%, and the grain size of 150 micrometers or less] was corned into granulation with a diameter [ of 3mm ], and a die length of 3mm, and this granulated body was put into the electric furnace, and from a room temperature to 780 degrees C, it heated by part for programming-rate/of 1 degree C, and while amounting to 780 degrees C, it took out from the electric furnace and cooled radiationally. The heating up time from 700 degrees C to 780 degrees C in this case was 80 minutes. CO<sub>2</sub> conversion of this thing was 95%, and specific surface area was 35.86m<sup>2</sup>/g.

[0027] The product made from example 3 - 6 calcium-hydroxide powder [Bell Woodwork Business, industrial use slaked lime

## BEST AVAILABLE COPY

95.9% [ of purity ] is classified to a fraction (45 micrometers or less, 45-75 micrometers, 75-150 micrometers, and 150-300 micrometers). 25 % of the weight of water was added to the powder of each fraction, it corned using disk PERETA [Fuji Paudal Make] into granulation with a diameter [ of 3mm ], and a die length of 3-4mm, this granulated body was heated to 700 degrees C by part for programming-rate/of 10 degrees C, and, subsequently it calcinated by whenever [ stoving temperature / of 900 degrees C ] for 1 hour. Thus, about the obtained calcium-oxide porous body, CO<sub>2</sub> conversion and specific surface area were measured. This result is shown in Table 1.

[0028]

[Table 1]

試 料	フラクションの粒度	比表面積 (m <sup>2</sup> / g)	CO <sub>2</sub> 反応率 (%)
実施例 3	45 μm 以下	20.02	92.53
実施例 4	45 ~ 75 μm	13.86	95.71
実施例 5	75 ~ 150 μm	8.92	93.58
実施例 6	150 ~ 300 μm	7.66	91.18

[0029] Each calcium-hydroxide powder with a particle size of 300 micrometers or less gives the calcium oxide more than specific-surface-area of 5m<sup>2</sup>/g so that clearly from this table.

[0030] After corning the calcium hydroxide used in the example 7 example 1 in the diameter of 3mm, and die length of 3mm, it put into the electric furnace, heated to 700 degrees C by part for programming-rate/of 10 degrees C, and calcinated for 30 minutes at this temperature. The heating up time from 390 degrees C to 470 degrees C in this case was 8 minutes. Thus, pore distribution of the obtained calcium-oxide porous body was measured. Consequently, it turned out that this invention article has the pore of the range of 0.02-0.2 micrometers.

[0031] The same calcium-hydroxide powder as what was used in examples 8-13 and the example of comparison 1 example 6 was corned in the diameter of 3mm, and die length of 3mm, was heated to the temperature from which it differs between 400-1100 degrees C from a room temperature by part for programming-rate/of 10 degrees C with an electric furnace, and was calcinated for 30 minutes each. The heating up time from 390 degrees C to 470 degrees C in this case was 8 minutes. Thus, CO<sub>2</sub> conversion and specific surface area of a calcium-oxide porous body which were obtained are measured, and the result is shown in Table 2.

[0032]

[Table 2]

試 料	最終加熱温度 (°C)	比表面積 (m <sup>2</sup> / g)	CO <sub>2</sub> 反応率 (%)
実施例 8	500	21.41	93.17
実施例 9	600	23.68	94.35
実施例 10	700	18.53	97.18
実施例 11	800	15.84	96.42
実施例 12	900	10.41	96.07
実施例 13	950	6.97	92.54
比較例 1	1100	3.50	52.34

[0033] what was used in examples 14-16 and the example of comparison 2 example 2 -- the same -- calcium-carbonate powder with a grain size of 150 micrometers or less was corned in the diameter of 3mm, and die length of 3-4mm, this granulated body was inserted in the electric furnace, the temperature up was carried out from the room temperature the rate for 10-degree-C/to the temperature of 800 degrees C, 900 degrees C, 950 degrees C, and 1100 degrees C, respectively, and it calcinated for 30 minutes after reaching a maximum temperature. Subsequently, each sample was taken out from the electric furnace, it cooled, and the calcium-oxide porous body was obtained. CO<sub>2</sub> conversion and specific surface area of this thing are measured, and that result is shown in Table 3.

[0034]

[Table 3]

試 料	最高加熱温度 (°C)	比表面積 (m <sup>2</sup> / g)	CO <sub>2</sub> 反応率 (%)
実施例 14	800	8.54	93.89
実施例 15	900	12.72	97.00
実施例 16	950	10.08	95.05
比較例 2	1100	4.62	65.21

## BEST AVAILABLE COPY

---

[Translation done.]

# BEST AVAILABLE COPY

## \* NOTICES \*

JPO and NCIP are not responsible for any damages caused by the use of this translation.

- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

---

## CORRECTION OR AMENDMENT

---

[Kind of official gazette] Printing of amendment by the convention of 2 of Article 17 of Patent Law  
 [Section partition] The 1st partition of the 3rd section  
 [Publication date] February 20, Heisei 13 (2001. 2.20)

[Publication No.] JP,7-149580,A  
 [Date of Publication] June 13, Heisei 7 (1995. 6.13)  
 [Annual volume number] Open patent official report 7-1496  
 [Application number] Japanese Patent Application No. 4-345159  
 [The 7th edition of International Patent Classification]

C04B 38/00 303  
 B01J 20/04  
 C01F 11/04  
 11/06

[FI]

C04B 38/00 303 Z  
 B01J 20/04 A  
 C01F 11/04  
 11/06

[Procedure revision]  
 [Filing Date] November 30, Heisei 11 (1999. 11.30)  
 [Procedure amendment 1]  
 [Document to be Amended] Specification  
 [Item(s) to be Amended] 0014  
 [Method of Amendment] Modification  
 [Proposed Amendment]  
 [0014] Although it is necessary to continue a temperature up further in order to aim at perfect baking of a brewing raw material, it is the temperature which does not exceed 950 degrees C in this case, and it must take care that CO<sub>2</sub> conversion moreover does not become 40% or less in the case of extensive processing, and it must be performed. This extracts a sample for every fixed time amount progress, and is performed by measuring CO<sub>2</sub> conversion about the sample.

[Procedure amendment 2]  
 [Document to be Amended] Specification  
 [Item(s) to be Amended] 0019  
 [Method of Amendment] Modification  
 [Proposed Amendment]  
 [0019]  
 [Effect of the Invention] Since the calcium oxide of this invention has a large specific surface area of at least 5m<sup>2</sup>/g, shows very high activity above and is a porous body, since it can use for a catalyst, the adsorbent for exhaust gas, the heat insulating material for steel manufacture, the flux for steel manufacture, etc., it has the advantage of being very easy to deal with it, as a product of a larger grain size than the conventional calcium oxide. Moreover, according to this invention approach, there is also an advantage that a calcium oxide with the activity according to the purpose of use can be obtained by selection of manufacture conditions.

[Procedure amendment 3]  
 [Document to be Amended] Specification  
 [Item(s) to be Amended] 0021  
 [Method of Amendment] Modification  
 [Proposed Amendment]  
 [0021]  
 [Equation 1]

**BEST AVAILABLE COPY**

$$\text{CO}_2 \text{ 反応率 (\%)} = \frac{\text{反応前 CO}_2 \text{ 濃度 (\%)} - \text{反応後 CO}_2 \text{ 濃度 (\%)}}{\text{反応前 CO}_2 \text{ 濃度 (\%)}}$$

[Procedure amendment 4]

[Document to be Amended] Specification

[Item(s) to be Amended] 0030

[Method of Amendment] Modification

[Proposed Amendment]

[0030] Example 7

After corning the calcium-hydroxide powder used in the example 1 in the diameter of 3mm, and die length of 3mm, it put into the electric furnace, heated to 700 degrees C by part for programming-rate/of 10 degrees C, and calcinated for 30 minutes at this temperature. The heating up time from 390 degrees C to 470 degrees C in this case was 8 minutes. Thus, pore distribution of the obtained calcium-oxide porous body was measured. Consequently, it turned out that this invention article has the pore of the range of 0.02-0.2 micrometers.

---

[Translation done.]